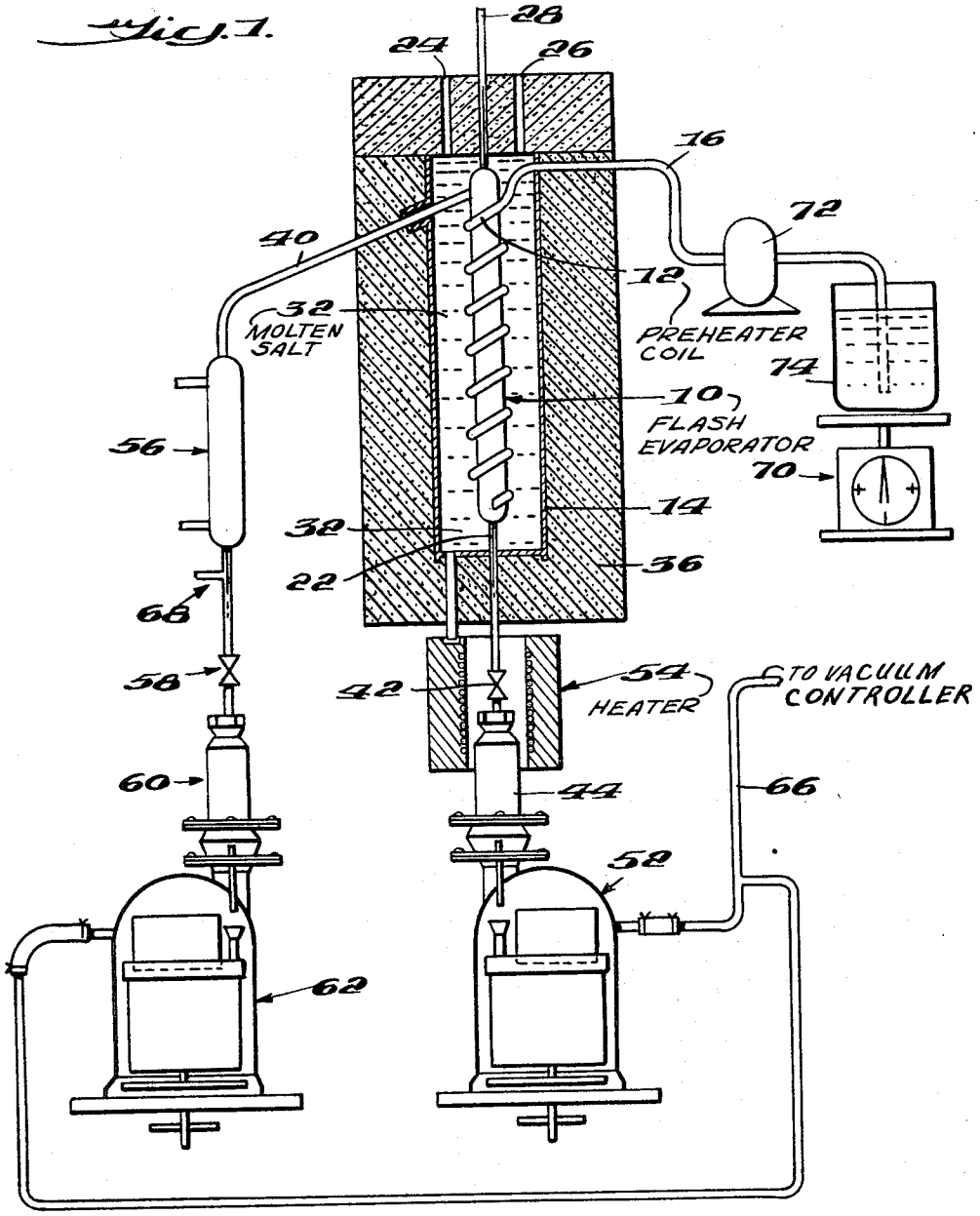
Aug. 27, 1968 J. J. DU BOIS ET AL 3,399,116

LABORATORY-SCALE FLASH STILL FOR PETROLEUM OIL FRACTIONS

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Fig. 1.



INVENTOR
Jule J. DuBois
John A. Glover
Robert F. Buehler

BY McLean, Morton & Boustead
ATTORNEY

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Fig. 2.

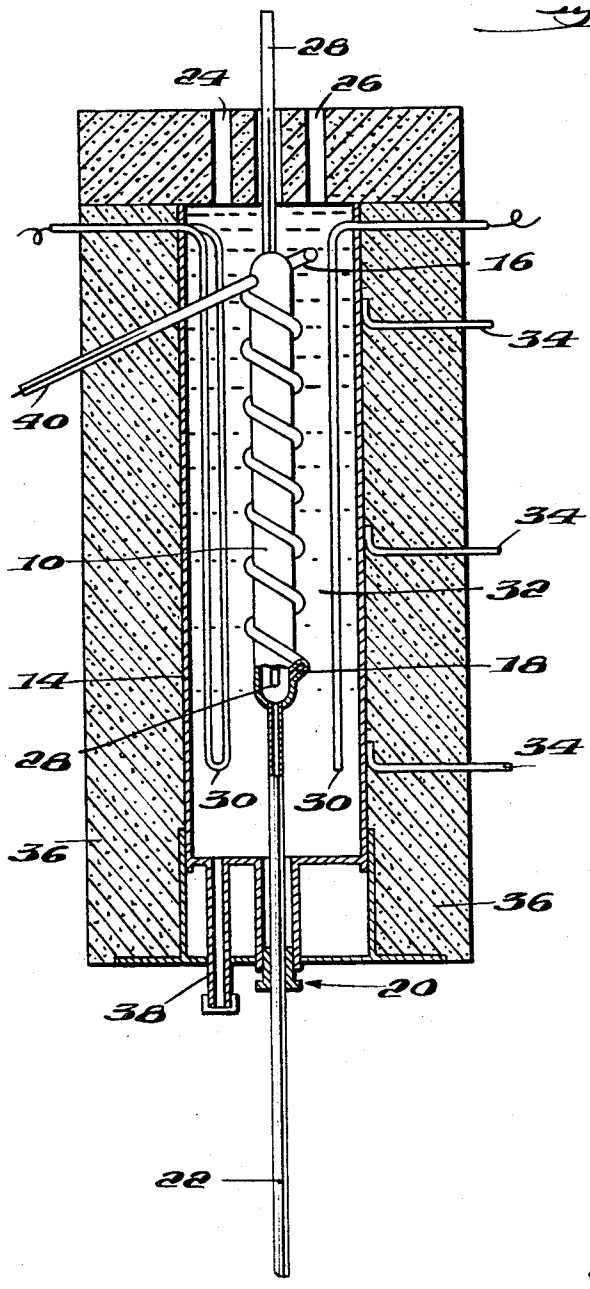


Fig. 3.

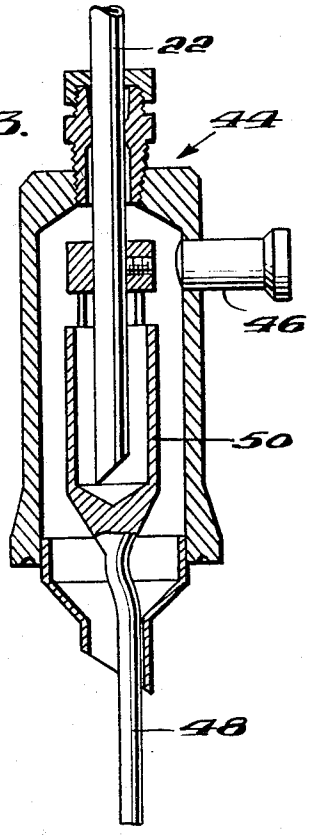
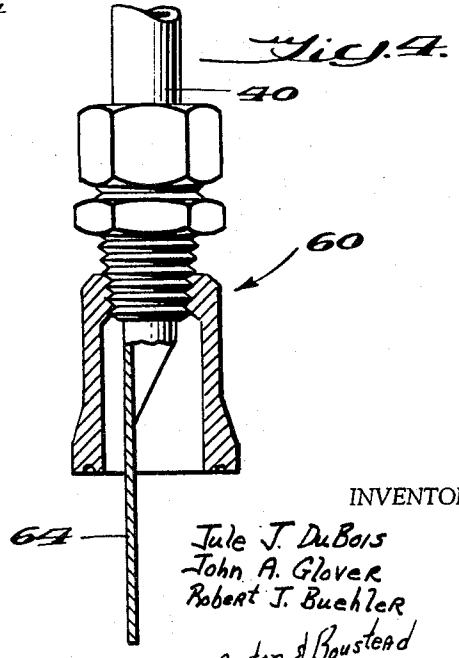


Fig. 4.



INVENTOR

Jule J. DuBois
John A. Glover
Robert J. Buehler

McLean, Morton & Boustead

ATTORNEY

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3,399,116

LABORATORY-SCALE FLASH STILL FOR PETROLEUM OIL FRACTIONS

Jule J. Du Bois, Schenectady, N.Y., and John A. Glover, Munster, and Robert J. Buehler, Whiting, Ind., assignors to Sinclair Research, Inc., New York, N.Y., a corporation of Delaware

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ABSTRACT OF THE DISCLOSURE

A laboratory scale flash still, employing a preheating coil surrounding a flash chamber with dual removing means, the preheating coil and flash chamber being substantially immersed in a molten salt heating medium, and means for maintaining the dual removing means at equal subatmospheric pressures.

This invention relates to a laboratory scale flash distillation still of improved utility in flashing such materials as reduced crudes, heavy residues, and penetration asphalts. More particularly, this invention concerns a laboratory scale flash distillation still which can be used to produce petroleum products of the quality generally expected from commercial operations.

For a long time there has been a need for a continuous laboratory scale equilibrium flash distillation still which can be used for processing various heavy hydrocarbon crudes in performing general assay work. Such a still is particularly necessary because bench scale reactor studies do not produce products in large enough volume to be utilized in the pilot plant scale flash distillation still. Some of the problems which have acted as a deterrent to the construction of such a still have been the overall poor operability of previously constructed small stills and a general reluctance to operate in a distillation laboratory a still which utilizes a mercury vapor heating jacket.

An improved laboratory scale equilibrium flash distillation still has now been developed which has eliminated the operability problems and those associated with the use of a mercury vapor heating means. Thus, the improved flash distillation still of the present invention can be used to process various crude hydrocarbons in producing petroleum asphalts and corresponding vacuum gas oils of the quality normally expected from only commercial operations. Further, by substituting a molten salt bath for the more hazardous mercury vapor heating means used by existing laboratory stills, the flash still of the present invention can be operated in distillation laboratories without the problems and dangers associated with mercury vapor heating.

The equilibrium flash still of the present invention broadly comprises a flash zone, a preheating zone and a heating zone, with the preheating zone surrounding the flash zone and the preheating zone and flash zone being disposed within the heating zone; means for introducing a heat transfer medium into the heating zone, means for removing the heat transfer medium from the heating zone, means for heating the heat transfer medium in the heating zone, means for introducing feed sequentially into the preheating zone and then into the flash zone, a first removing means for recovering the light end product from the flash zone, a second removing means for recovering the heavy end product from the flash zone and means for maintaining the first and second removing means at an equalized subatmospheric pressure.

The present invention will be more fully understood from the detailed description hereinbelow and the accompanying drawings wherein—

FIGURE 1 shows a distillation apparatus incorporating the still of this invention;

FIGURE 2 is a sectional elevational view of the still;

FIGURE 3 illustrates details of the bottoms adapter for the distillation apparatus; and

FIGURE 4 illustrates details of the overhead adapters.

More specifically, the equilibrium flash still of the present invention comprises a flash chamber **10**, a preheating coil **12**, and a heating vessel **14** with flash chamber **10** and preheating coil **12** being arranged within the heating vessel. Preheating coil **12** consists of tubing which is wound around, and in contact with, flash chamber **10** from the upper to the lower portion thereof. Feed material passes from the outside connection **16** of coil **12** through the coil **12** which serves to preheat the feed. The preheated feed is then introduced into the flash chamber **10** tangentially at a point slightly above the bottom portion thereof through opening **18** (see FIGURE 2). Heating vessel **14** surrounds flash chamber **10** for substantially its entire length and is provided with closures at its bottom and its top. The closure at the bottom includes a packed gland exit opening **20** which has a bottoms recovery line **22** extending therethrough. The closure at the top is removable and includes a fill port **24** through which the heat transfer medium **32** can be introduced into the heating vessel **14**, a stirrer port **26** and a thermowell **28**. Stirrer port **26** is provided so that the heat transfer medium **32** can be gently agitated by a stirrer (not shown) to maintain uniform temperatures through the entire bath. Thermowell **28** is provided to permit measurement of the temperature gradient in flash chamber **10**. Electric immersion heaters **30** are arranged in vessel **14** to supply the heat to maintain the heat transfer medium in a molten state. Heaters **30** are powered by an electric source which can supply sufficient electric power to heat the heat transfer medium, e.g., up to about 400 watts per heater, which source is not shown. The heat transfer medium **32** used in the present invention is, for example, a molten salt bath, such as sodium or potassium salts. Skin thermocouples **34** are spaced on the outside surface and along the entire length of the heating vessel **14** to furnish temperature data for temperature control. Several inches of insulation **36** on vessel **14** serve to limit heat loss from the vessel. A drain **38** is provided in vessel **14** for drawing off the heat transfer medium when desired, such as when the apparatus is not being used.

Flash chamber **10** has in addition to a bottoms recovery line **22**, an overhead vapor line **40**. A shut-off valve **42** is located in the bottoms recovery line **22** which line then enters a liquid seal device **50** within bottoms adapter, generally designated by numeral **44**, which is provided with a plugged access port **46** and drip rod **48**. The bottoms pass through line **22** into the seal device **50**, i.e., an overflow cup, and then to receiver assembly **52**. The bottoms line **22** and shut-off valve **42** are enclosed by an electric heater **54** which insures optimum heat control and sample protection.

Overhead vapor from flash chamber **10** passes through vapor line **40**, condenser **56**, e.g., air or water cooled, and a quick-opening diaphragm valve **58** into an overhead receiver assembly, i.e., overhead adapter **60**. Overhead adapter **60** is attached to overhead receiver **62** and includes a drip rod **64** which extends into the overhead receiver. The overhead product passes from overhead adapter **60** to the overhead product receiver assembly **62** which is identical with the bottoms product receiver assembly **52**. A vacuum controller **66** maintains the overhead vapor line **40** and bottoms recovery line **22** at equalized subatmospheric pressures and provides satisfactory pressure stability down to about 1 mm. Hg. Still pressure is measured through a tap **68** in the overhead line **40** below the condenser **56** by a pressure measuring device (not shown) such as a Dubrovin gauge a closed end mercury

manometer or, at higher pressures, by an open end mercury manometer. Suitable manometers indicate the pressure at each receiver for control of pump-out pressures or differential settings. An adjustable liquid level bubbler may be used to provide a pressure differential between the overhead and bottoms receivers, thus providing control on the liquid level within the flash chamber. However, this unit can be operated without this device, and although the liquid level may be below the heating vessel, there is no evidence of deterioration in product quality. Operation of the equilibrium flash still is as follows: Heat transfer medium, e.g., salt, is introduced through fill port **24** until flash chamber **10** is substantially surrounded. Electric immersion heaters **30** are then turned on and the medium heated to the desired operating temperature. Flash chamber **10** is then evacuated to the desired operating pressure and the feed, which is measured by scale **70**, is pumped **72** from tank **74** through line **16** into preheat coil **12** to be introduced into the lower portion of the flash chamber at inlet **18**. The feed is cracked into the resulting products in chamber **10**.

Feed rates of about 50 to 200 grams per hour are generally used in the flash still and upon introduction of the feed into the bottom of the flash chamber **10**, the vapor liquid equilibrium is essentially complete. The flash still is generally run at a temperature of about 680° F. to 740° F. Temperature control is not a problem in the distillation apparatus of the present invention and thus when using the heat transfer medium, e.g., molten salt bath, temperatures of up to 800° F. and even higher are not considered excessive in this type of unit.

The flash still has proved to be very valuable in that it can be used for a variety of feedstocks such as reduced crudes, heavy residues, penetration asphalt, and even feedstocks yielding zero penetration bottoms, which can be handled by the use of auxiliary heat sources such as infrared lamps. These lamps can be assembled promptly when needed and removed when the condition warrants. The still has also proven to be very useful in crude assay work, producing petroleum asphalt and corresponding vacuum gas oils of the qualities to be expected from commercial operations. Example I demonstrates such use.

EXAMPLE I

A mid-continent crude is fractionated on a glass true-boiling-point still yielding cuts to a nominal 400° F. end point. The crude is further reduced on the same still under vacuum, taking cuts to a 650° F. atmospheric boiling point. The vacuum bottoms (35.6 volume percent on crude) are next flashed in the flash still of the present invention at 680° F. Several runs are made at various pressures. The flash chamber conditions, along with the feed, gas oil and bottoms yield and quality measurements are listed in Table I.

TABLE I.—MIDGET STILL VACUUM FLASH DISTILLATION OF 35.6 VOL. PERCENT MID-CONTINENT REDUCED CRUDE

	Reduced Crude	Gas Oil Overhead				
Pressure, mm. Hg		1.0	2.0	4.0	6.0	8.0
Temperature, ° F		680	680	680	680	680
Liquid, percent on crude	35.56	25.82	25.15	24.34	23.27	22.66
Gravity, ° API	20.6	24.8	24.9	25.3	25.4	25.5
Color (Saybolt)		B-16	B-16	B-16	B-16	B-16
Con. carbon, wt. percent	6.25	.948	.726	.552	.500	.390
V/100, SSU	3003					
V/130, SSU	988	225.9	210	194.2	181.4	168.6
V/210, SSU	139.9	60.0	58.8	55.9	54.1	53.4
Mol. wt	596	424	420	415	400	401
Pour, ° F	+75	+95	+100	+95	+100	+95
Flash, COC	465	435	450	445	445	435
Aniline Point, ° C	98.6	93.6	94.2	93.9	93.3	93.3
Sulfur, wt. percent	.851	.719	.701	.688	.648	.615
Nickel, p.p.m	22	2		2		
Vanadium, p.p.m	39	2		2		

	Asphalt or Tar				
Pressure, mm. Hg	1.0	2.0	4.0	6.0	8.0
Temperature, ° F	680	680	680	680	680
Liquid, percent on crude	9.74	10.41	11.22	12.29	12.90
Sp. Gr. 77/77° F	.9905	.9887	.9846	.9782	.9773
Pen. at 77° F	107	161	245	Soft	Soft
V/275 SSF	160.6	119.7	94.3	75.8	62.9
Oliensis, A.O	Neg.	Neg.	Neg.	Neg.	Neg.
Oliensis, 24 hours	Neg.	Neg.	Neg.	Neg.	Neg.
R & B soft. pt., ° F	114	106	99	92.5	88.5
Sulfur, wt. percent		1.25			
Ductility	100+	100+	100+	+100	+100
V/210 SSF		902.2			
Nickel, p.p.m		62			
Vanadium, p.p.m		108			

EXAMPLE II

To further illustrate the versatility of the still, a severely cracked residual is flash distilled at two different sets of conditions. Yields and product quality measures on the gas oils and asphalt bottoms are listed in Table II. The high viscosity and low penetration of bottoms produced are of particular interest from the standpoint of the operability of the still.

TABLE II.—MIDGET STILL VACUUM FLASH DISTILLATION OF A SEVERELY CRACKED RESIDUAL OIL

Flash temperature, ° F	740	678
Flash pressure, mm. Hg	30	10
Gas oil overhead:		
Yield, wt. percent of feed	47.0	40.0
Gravity, ° API	19.6	19.6
Carbon res., rams	1.248	0.951
Wt. percent:		
Carbon	86.83	86.59
Hydrogen	11.71	11.72
Sulfur	0.771	0.727
Nitrogen	0.34	0.35
Mole wt	399	392
Tar Bottoms:		
Yield, wt. percent of feed	53.0	60.0
Gravity, ° API	1.0	2.8
Carbon res., con	35.74	32.14
NC_5 insolubles, wt. percent	28.66	22.54
Furol vis. at 275° F		213.0
Penetration, 77° F	5	12

It is claimed:

1. A laboratory scale flash still for separation of a petroleum fraction which comprises a preheating coil and a vertically disposed flash chamber, said preheating coil being wound externally about said chamber from the upper to the lower end thereof and interconnecting at its lower end with the interior lower section of said flash chamber for delivering the preheated petroleum fraction thereto for flash distillation separation of said fraction, an enclosed heating vessel substantially surrounding said preheating coil and flash chamber, said heating vessel containing a molten salt heat transfer medium and a heating means in said heat transfer medium for maintaining said medium at a predetermined temperature, a charge pipe for introducing the petroleum fraction feed

into said preheating coil at its upper end an overhead recovery means near the top of said flash chamber for removing the light end product from said flash chamber, a bottoms recovery means near the bottom of said flash chamber for removing the heavy end product from said flash chamber, and a vacuum controller means for maintaining both of said recovery means at equalized subatmospheric pressures.

2. The still of claim 1 wherein said flash chamber comprises an elongated flash chamber.

3. The still of claim 1 including insulating means surrounding said heating vessel.

4. The still of claim 3 wherein said bottoms recovery means includes a seal means comprising an overflow cup, and a bottoms recovery line interconnecting the bottom of said flash chamber and the interior of said overflow cup.

5. The still of claim 4 wherein said bottoms recovery means includes a receiver and said overflow cup has a drip rod thereon adapted to pass liquid from said cup to said receiver.

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NORMAN YUDKOFF, *Primary Examiner.*

F. E. DRUMMOND, *Assistant Examiner.*